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A STUDY OF THE SOLIDIFICATION PARAMETERS INFLUENCING STRUCTURE AND PROPERTIES IN MAR-M246 (Hf)

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16. ABSTRACT

The nickel-base superalloy MAR-M246(Hf) was studied to determine the factors affecting basic morphology and fatigue properties. Of particular interest was the degradation of fatigue properties with deviation from the [001] growth orientation. Examination of directionally solidified samples showed a dependence of carbide shape and interdendritic segregation on growth rate. Heat treatment studies focused on the gamma prime structure, determining that it reaches maximum growth after twenty-four hours but its size and stability depends on the temperature of the treatment. Fatigue test specimens were oriented crystallographically in the failed and unfailed regions and found to have rotated their orientation during the testing if they were located a significant distance from [001]. This would place increased strain on the crystal and precipitate early failure.

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TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS (Concluded)

TECHNICAL MEMORANDUM

A STUDY OF THE SOLIDIFICATION PARAMETERS INFLUENCING STRUCTURE AND PROPERTIES IN MAR-M246 (Hf)

BACKGROUND AND PURPOSE

Superalloys are alloys (usually nickel-based) that have high strength and good corrosion resistance at elevated temperatures up to 1400°C. Their uses range from petrochemical equipment to aircraft, marine, industrial, and vehicular gas turbines. Since these are high-technology materials with high temperature applications there is a constant push from industry for their improvement. A few degrees increase in engine operating temperature means an even greater increase in the efficiency (with concurrent decrease in fuel consumption of the engine) [1].

In order to meet the demands of industry, the superalloys have developed into complex combinations of materials. They usually average around ten constituents. For example MAR-M246(Hf), the superalloy in the Shuttle Main Engine contains nickel, chromium, cobalt, molybdenum, tungsten, tantalum, aluminum, titanium, carbon, boron, zirconium, and hafnium. Table 1 lists the known contributions of these different additions. All constituents combine to form three types of phases in the alloy structure: the matrix (γ), the gamma prime (γ'), and carbides. Figure 1 gives examples of these phases.

The matrix phase is a nickel-based FCC austenite which contains solid solution strengthening elements. Some of the additions (e.g., Mo and W) are added specifically to provide strength at high temperatures. The gamma prime has a complex ordered structure which precipitates coherently with the matrix to provide precipitation hardening. The mechanical properties of the superalloy can usually be related to the shape, size, and amount of gamma prime present. There are three types of carbides which can form: MC, M_6C, and $M_{23}C_6$. The influence of these carbides on the properties depends on their chemical formula, shape, and location [2]. If distributed in the grain boundary or as blocky particles in the matrix, they are sometimes considered helpful. However, it should be pointed out that the real influence (harmful versus helpful) of the carbides is still a debated issue.

The present program in the Materials and Processes Laboratory on MAR-M246(Hf) was aimed at studying the influence of solidification parameters on the structure and fatigue properties. It has been noted in earlier property tests [3] that the fatigue life decreased as the crystallographic orientation deviated from [001]. Since this deviation could also cause variation in the segregation of the constituents as well as affect the dendrite arm spacings and dominant slip system, a multi-faceted program was begun to study these different phenomena.

APPROACH

In order to understand the property changes experienced by MAR-M246(Hf) undergoing fatigue tests, both solidification and the subsequent heat treatment were studied. In addition, single crystal specimens were fatigue tested, oriented using X-ray diffraction, and analyzed for segregation.

Samples were prepared for examination by mounting longitudinally and then polishing halfway into the cross-section using successively smaller grits. After etching with Adler's etchant the microstructure became visible. For volume fraction of carbide measurements using the Quantimet, the specimens were returned to the vibropolisher until all detail but the carbides was extinguished.

SOLIDIFICATION

A schematic of the directional solidification system is shown in Figure 2. Details on the directional furnace have been supplied elsewhere [4]. A series of samples was directionally solidified at different rates to obtain dendrite arm spacings, carbide volume fraction, and shape dependence on growth parameters. Figure 3 shows the dependence of the arm spacings on growth rate. These follow the expected trend of arm spacing decreasing with growth rate. This effect is illustrated in Figure 4. The carbide shapes changed as a function of growth rate as can be seen in Figure 5. At slow rates the carbides appear blocky, while at high rates they adopt a script-like morphology. Quantitative analysis measurements were taken to determine the amount of carbides present in the samples. Figure 6 shows the dependence of volume percent carbides on the secondary arm spacings.

In order to determine if a deviation from the [001] had an effect on microstructure, polycrystalline samples were solidified. This insured that the grains measured had the same thermal history. The individual grains were oriented using X-ray diffraction and correlated with the primary arm spacings. Figure 7 shows the change in primary arm spacing with deviation from [001] for various directionally solidified samples. The arm spacings are decreasing with deviation from [001]. Figure 8 shows a typical polycrystalline cross-section and the accompanying grain orientation.

HEAT TREATMENTS

The alloy MAR-M246(Hf) is always heat treated prior to use. The standard heat treatment is 1221° ± 6°C, holding at heat for 2 hr ± 10 min, followed by cooling to room temperature then reheating in a vacuum or suitably protective atmosphere to 871° ± 14°C, holding at heat for 24 ± 1 hr, followed by cooling to room temperature and cleaning if required. The first is supposed to put all of the γ' back into solid solution in the matrix. The second part of the treatment controls the precipitation of γ'.

A series of temperature and times based on the standard heat treatment was selected for processing of a series of samples. Table 2 lists the selected heat treatments. Since the γ' structure can only be distinguished at higher magnification, replicas for the transmission electron microscope were made from each sample. Figure 9 shows the γ' structure from the standard heat treatment. Figures 10 through 13 show structures obtained from some of the other heat treat series. It appears that the γ' has not been fully solution heat treated at the 1175°C temperature and below. Measurements of the size and volume fraction of the γ' were taken using the Quantimet Image Analyzer. These results are plotted versus precipitation heat treating times in Figures 14 and 15. The amount of γ' levels off after 100 hr at 871°C and 100 hr at 982°C, but in the case of 982°C they are not stable, but continue to increase in size.

Microprobe traces were made across sets of three dendrite arms before and after selected heat treatments. Typical traces for some of the elements are given in Figure 16.

MECHANICAL TESTING

The MAR-M246(Hf) single crystals were fatigue tested in the tension-tension mode at 843°C until failure or run out ($>10^7$ cycles). The single crystals were screened by X-Ray Laue techniques and categorized into groups designated as 0, 5, 10, and 15 degrees off the [001] orientation. The off-axis orientation refers to the angle between the growth axis and [001]. "R" ratios varied from 0.4 to 0.8 for the series of samples (R refers to the minimum to maximum stress ratio). Figures 17 and 18 show the cycles to failure of the fatigue specimens for various R levels and crystallographic orientations. Although the crystals from the 15 degrees group tended to fail at lower number of cycles than the other groups, it was not a consistent effect for every crystal. After testing, the samples were sectioned parallel to the axis, mounted, polished, reoriented to determine their crystallographic orientations, and analyzed for dendrite arm spacing and carbide presence. The stereographic projection in Figure 19 shows the orientation of some of the tested specimens and their movement (if any) during testing.

SUMMARY AND CONCLUSIONS

As generally occurs, the dendrite arm spacings decreased with increasing growth rate. In addition, the finer the spacings, the smaller the amount of carbides present. Since deviation from [001] decreased the arm spacings for a given growth rate, the amount of carbides would be expected to decrease in a similar fashion, eliminating the possibility that an increase in harmful carbides accounts for lower fatigue properties in off-[001] axis crystals.

The carbide shapes can be controlled by controlling the growth rates. At fast rates, the carbides become script-like, forming networks for crack propagation. At slow rates they were blocky. Since changes in crystallographic orientation had no effect on the carbide shapes, an off-axis crystal would not be expected to fail prematurely because of a difference in the carbide morphology.

It is apparent from the microstructures that the γ' goes into solution between 1175°C and 1200°C, making it possible to decrease the present solution heat treatment temperature from 1221°C. The decrease would also help to prevent what appears to be melting of eutectic in some of the interdendritic areas. Since the amount of Hafnium has been found to reach as high as 18 percent in some of these regions, it is probable that this element is mainly responsible for the melting. As can be seen in Figure 16, while the solution heat treatment helps homogenize many of the elements in the alloy, it has minimal effect on the Hafnium.

As a result of the precipitation treatment, a steady-state amount of γ' phase is reached between 24 and 100 hr at 871°C. Extended periods do not increase the amount of γ' present, and tend to degenerate the structure. The size however remains relatively stable. The results of the study at 970°C indicate that at this temperature the amount of γ' reaches steady-state, but the size of the γ' is not stable. Therefore, even if the alloy is given the standard heat treatment and reaches its steady-state structure at 871°C, if it experiences higher temperatures, its structure will become unstable and eventually degenerate. The fatigue properties would then deteriorate.

Although it appears that time periods from 24 to 100 hr are desirable for precipitation of the desired γ' structure, it should be remembered that other phases, such as the carbides, are also affected by the heat treatment. Another research study is needed to determine the compromise treatment that will produce enough stable γ' without also precipitating harmful carbides.

The results of the fatigue tests indicate that when the crystallographic orientation is greater than 10 degrees from the [001] axis, the properties degenerate. Other investigators [5] recently studied the influence of orientation on the stress-rupture properties of MAR-M247 single crystals. For first-stage creep the slip system is $\{111\}$ $\langle 112 \rangle$. During second stage creep the $\{111\}$ $\langle 101 \rangle$ system becomes operative. The stress-rupture lives were influenced by the amount of lattice rotation required to produce intersecting slip. Once intersecting slip begins, the second stage of creep is entered and the material strain hardens. If a crystal was oriented such that large rotations were required to produce intersecting slip and second stage creep, the material experiences large strains and a shorter stress-rupture life.

Comparisons of several X-ray Laue photographs in the failed and undeformed regions of fatigue tested MAR-M246(Hf) crystals show that there was some rotation in these samples during testing. Similar to the stress-rupture test results, it is this rotation that eventually precipitates the failure. The further the sample has to rotate before reaching multiple slip conditions, the greater the probability for early failure. Since orientations located off-[001] axis satisfy these conditions, they therefore fail more easily.

TABLE 1. MAR-M246(Hf)

Element	Percent	Solid Soln. Strengthener	γ' Former	Carbide Former
Ni	60.0			
Cr	9.0	X		X
Co	10.0	X		
Mo	2.5	X		X
W	10.0	X		X
Ta	1.5		X	X
Al	5.5		X	
Ti	1.5		X	X
C	0.15			X
B	0.015			
Zr	0.05			
Hf	1.5			X

TABLE 2. HEAT TREATMENT NOMENCLATURES

	Solution Heat Treatment (2 Hours)				
	1150°C	1175°C	1200°C	1221°C	1250°C
No Further Treatment	MM-HT-150/1	MM-HT175/1	MM-HT200/1	MMHT221/1	MM-HT250/1
871°C/24 hrs	MM-HT-150/2	MM-HT175/2	MM-HT200/2	MM-HT221/2	MM-HT250/2
871°C/100 hrs	MM-HT150/4	MM-HT175/4	MM-HT200/4	MM-HT221/4	MM-HT250/4
982°C/24 hrs	MM-HT150/5	MM-HT175/5	MM-HT200/5	MM-HT221/5	MM-HT250/5
982°C/100 hrs	MM-HT150/6	MM-HT175/6	MM-HT200/6	MM-HT221/6	MM-HT250/6
1079°C/4 hrs 871°C/32 hrs	MM-HT150/7	MM-HT175/7	MM-HT200/7	MM-HT221/7	MM-HT250/7
871°C/433 hrs	MM-HT150/8	MM-HT175/8	MM-HT200/8	MM-HT221/8	MM-HT250/8
1150°C/24 hrs 1221°C/2 hrs 871°C/24 hrs				MM-HT221/9	
872°C/6 hrs	MM-HT150/10	MM-HT175/10	MM-HT200/10	MM-HT221/10	MM-HT250/10
982°C/500 hrs	MM-HT150/11	MM-HT175/11	MM-HT200/11	MM-HT221/11	MM-HT250/11

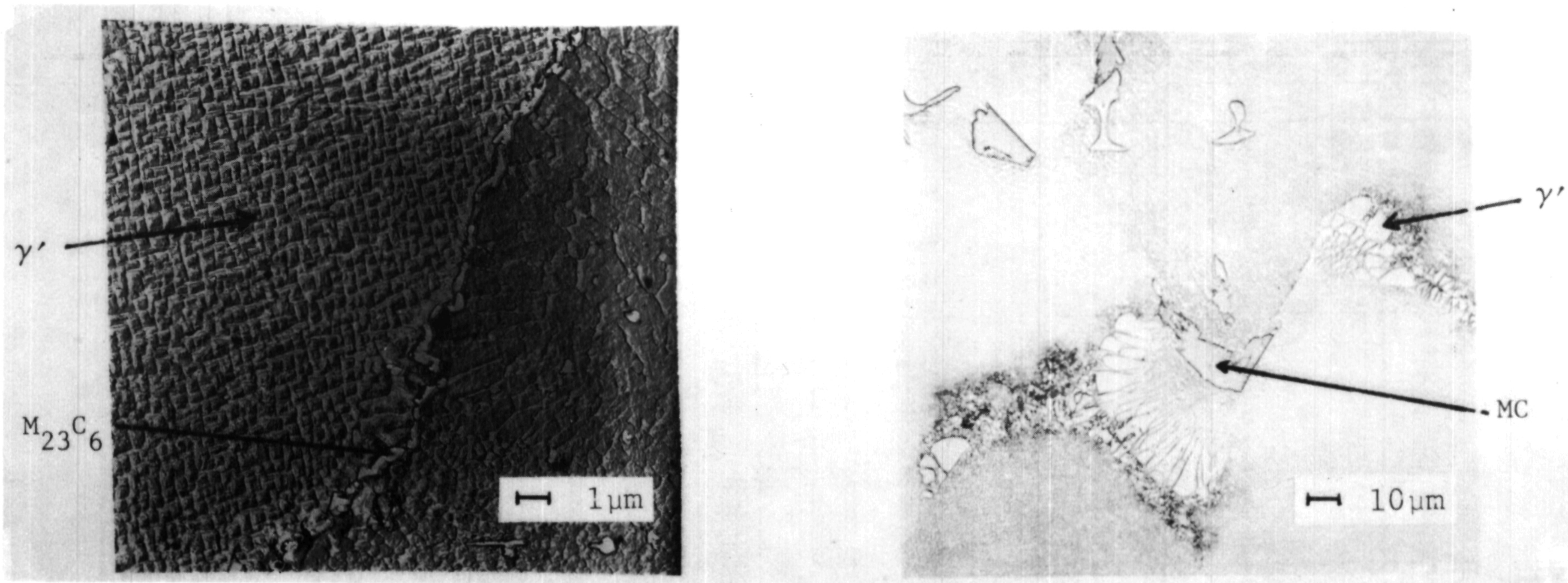


Figure 1. Microstructure of MAR-M-246(Hf).

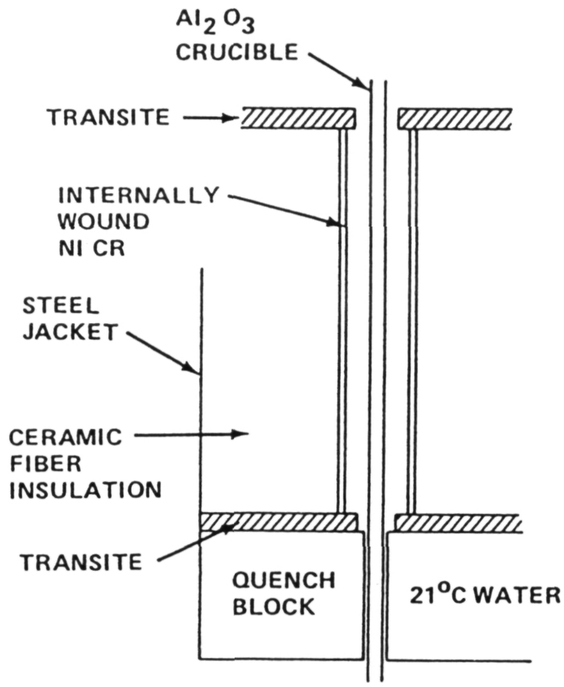


Figure 2. Schematic of directional solidification furnace.

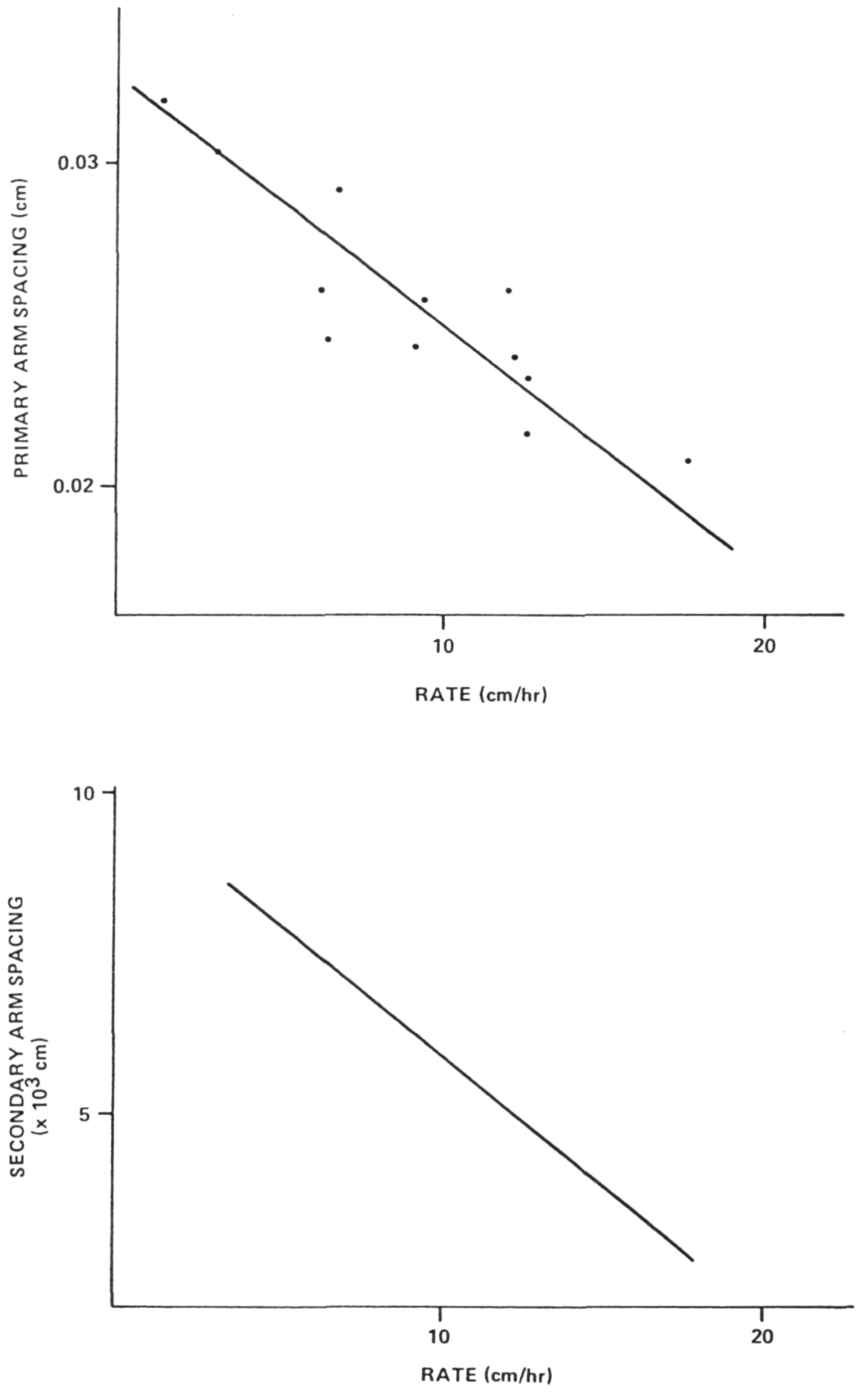
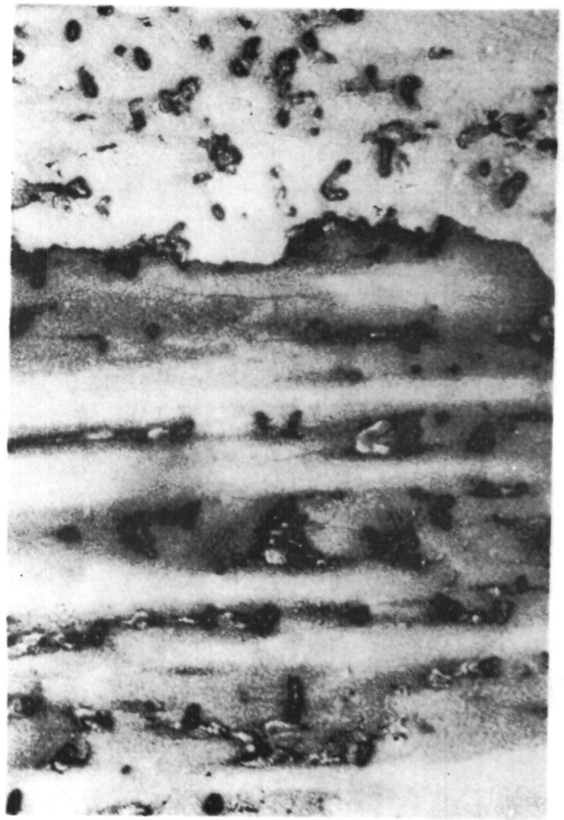


Figure 3. Primary and secondary arm spacings as a function of growth rate.

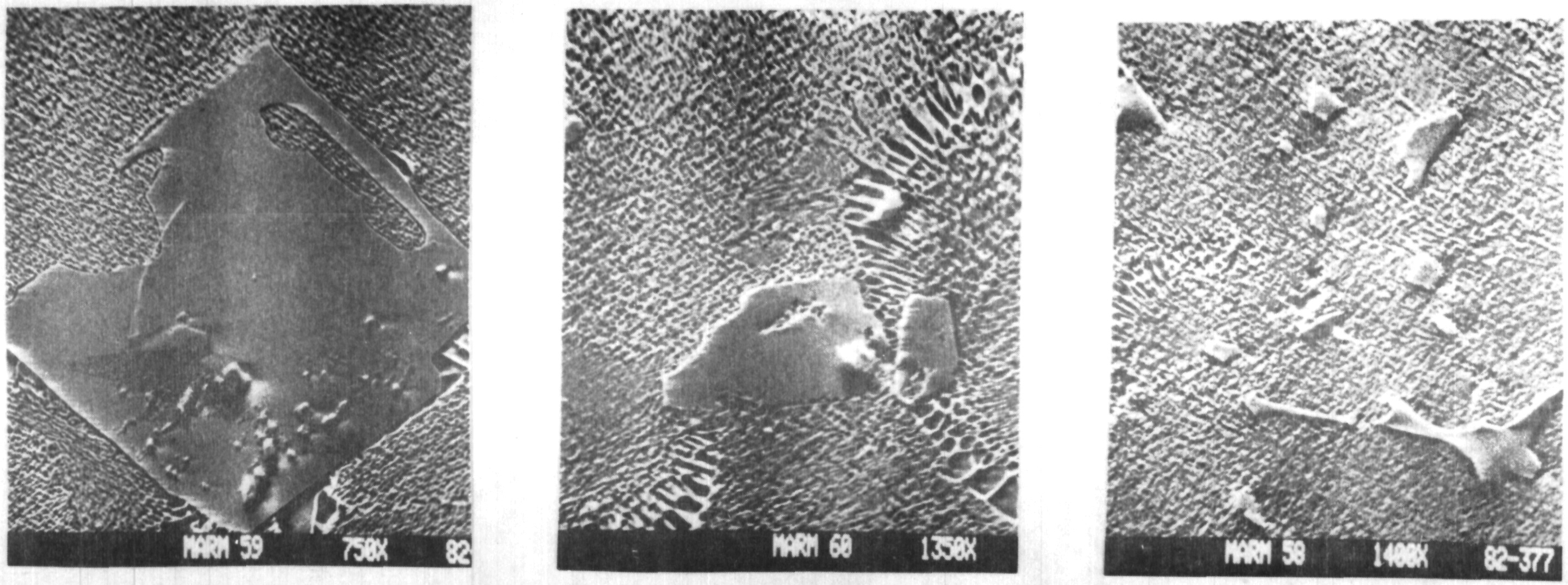


R = 1.74 cm/hour

R = 8.6 cm/hour

R = 42.6 cm/hour

Figure 4. Directionally solidified structures of MAR-M246(Hf), at different growth rates, G = 182°C/cm.



R=1.74cm/hr
G/R=1.8x10^5

R=4.5cm/hr
G/R=0.7x10^5

R=42.9cm/hr
G/R=0.07x10^5

Figure 5. Changes in carbide shape with growth rate, G = 87°C/cm, MAR-M246(Hf).

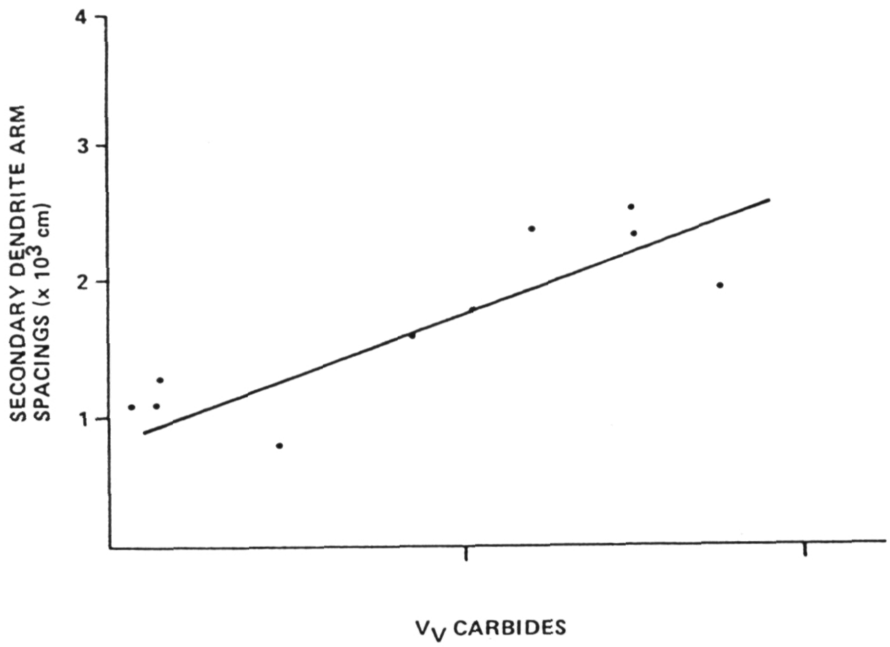


Figure 6. Volume fraction of carbides present as a function of secondary dendrite arm spacing.

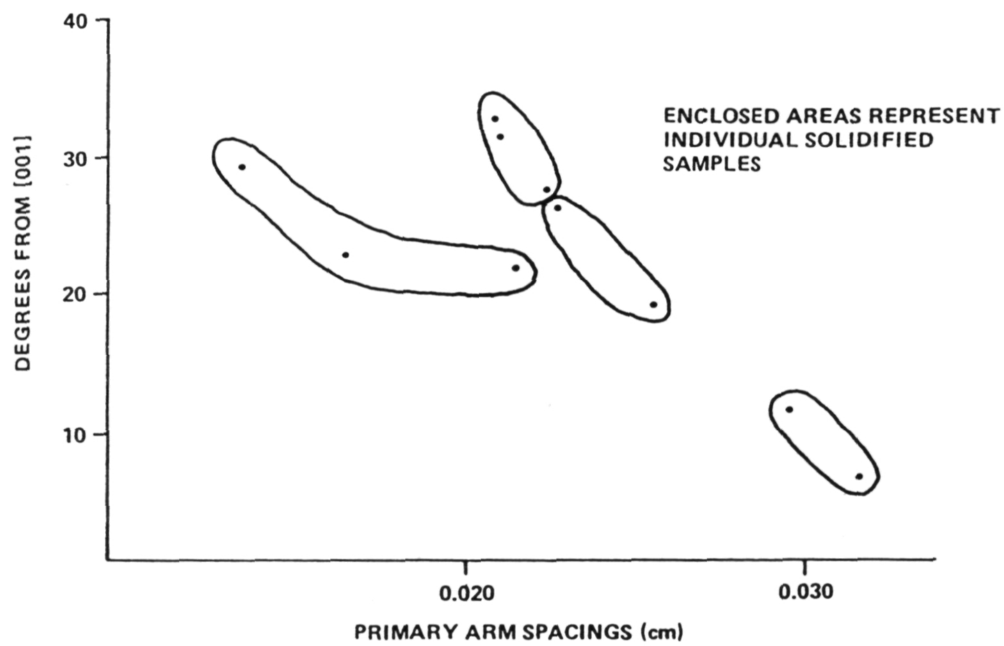


Figure 7. The variation of primary arm spacings with deviation from [001].

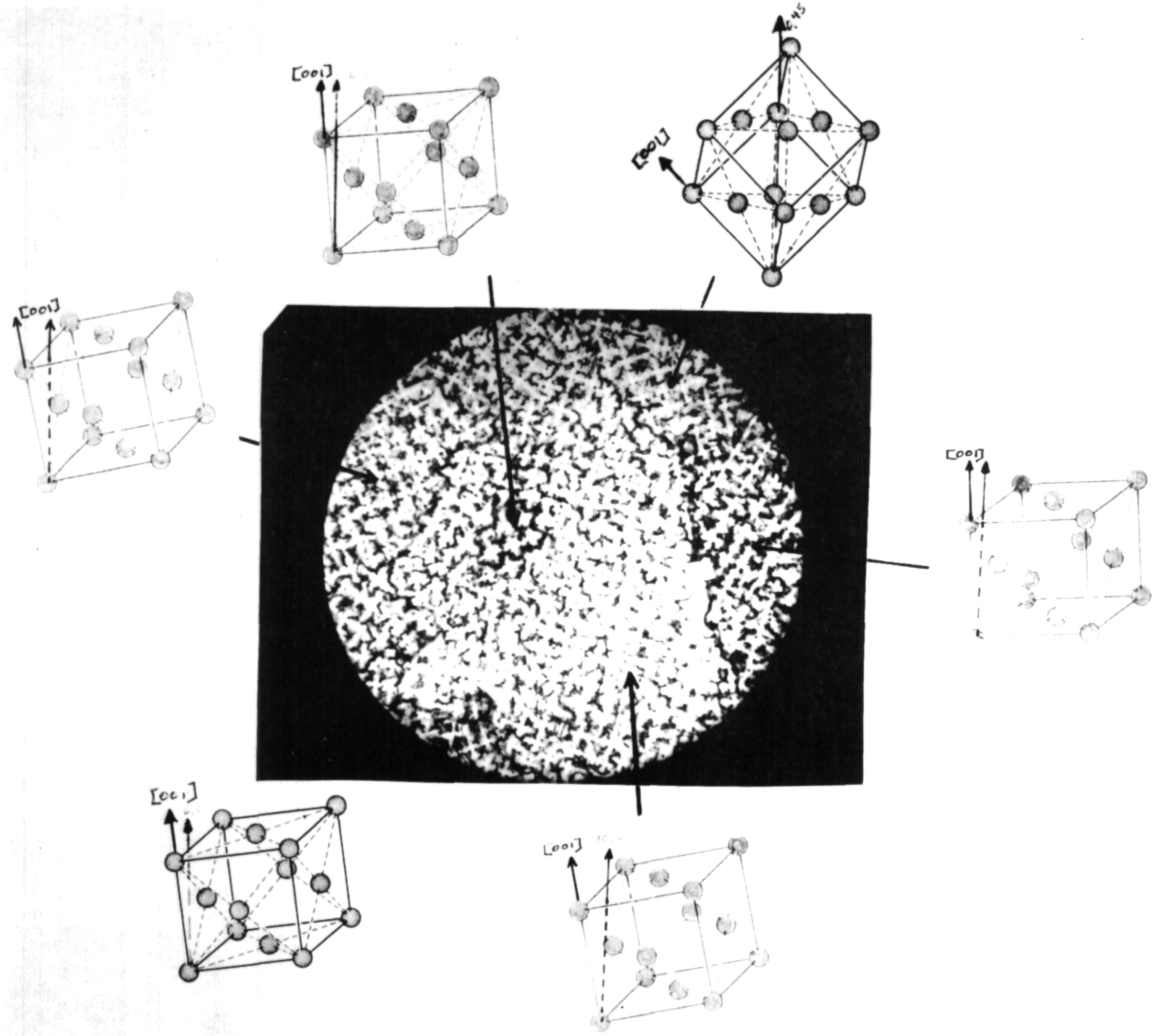


Figure 8. MAR M30 (bottom).

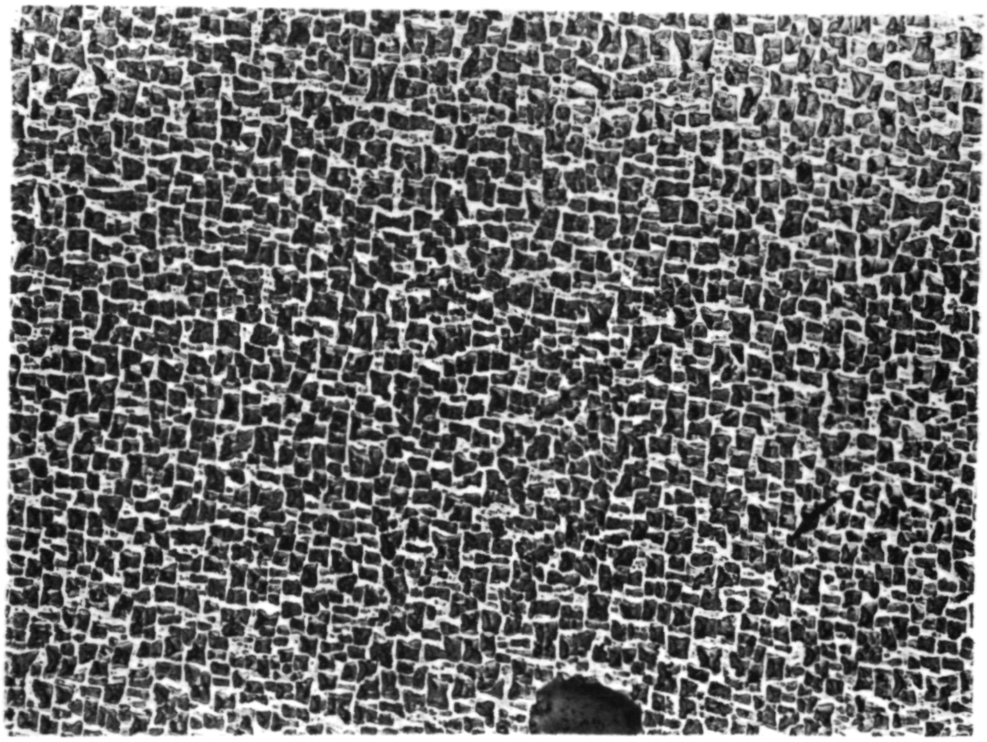


Figure 9. Transmission electron microscopy photograph of gamma prime structures in MAR-M246(Hf), 1150°C and 24 hr presolution heat treatment, 1221°C and 2 hr solution heat treatment, and 871°C and 24 hr precipitation heat treatment. (6000X)

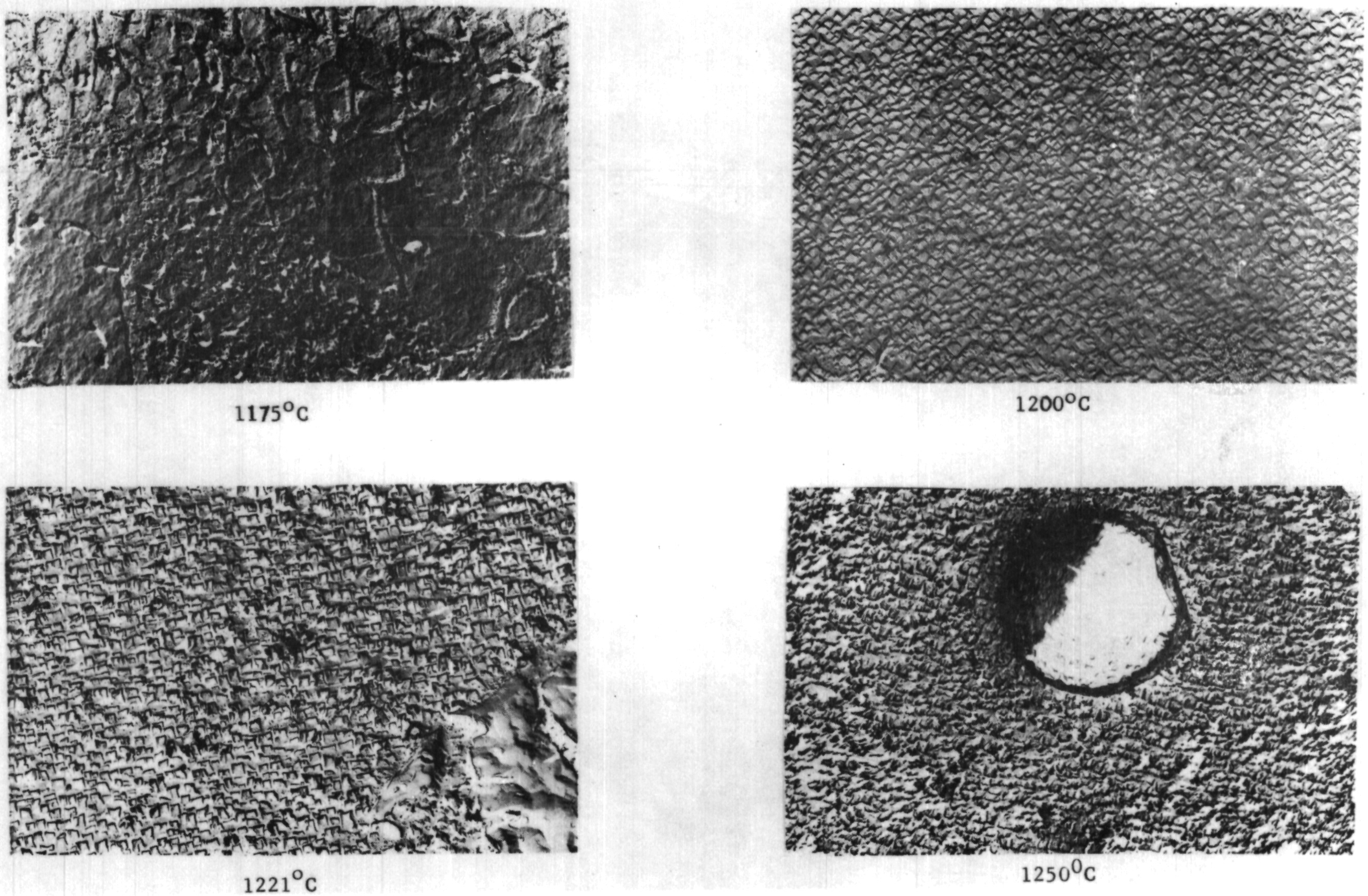
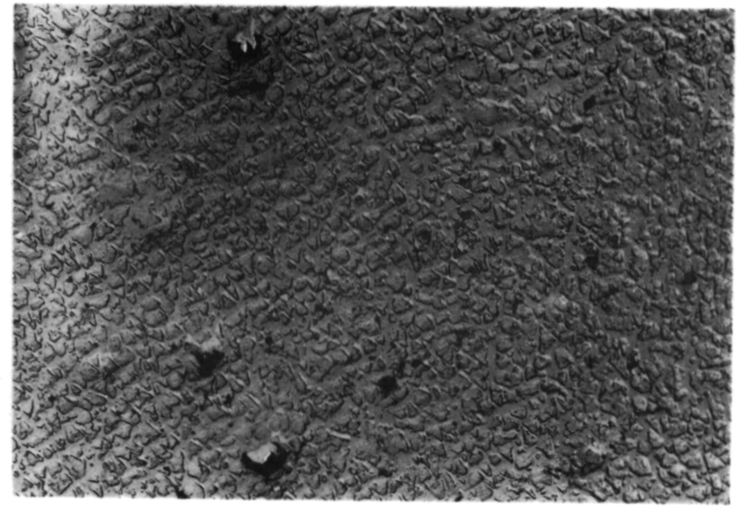
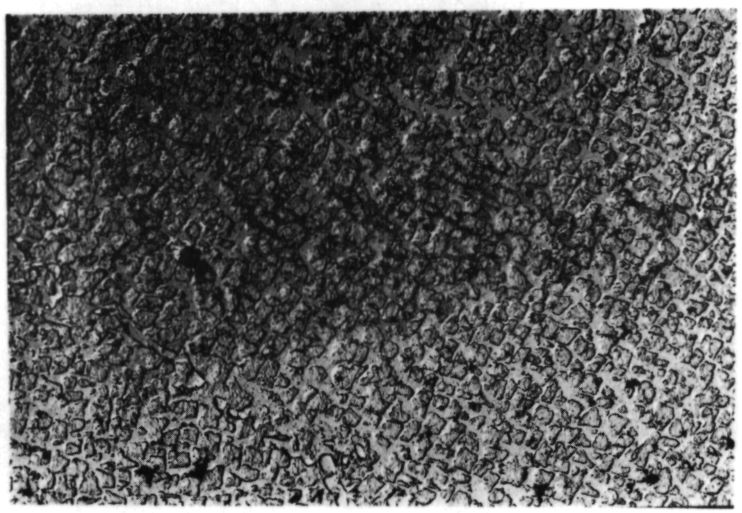


Figure 10. Transmission electron microscopy photographs of gamma prime structures in MAR-M246(Hf) at various solution heat treatment temperatures and 871°C, 24 hr precipitation heat treatment. (6000X)

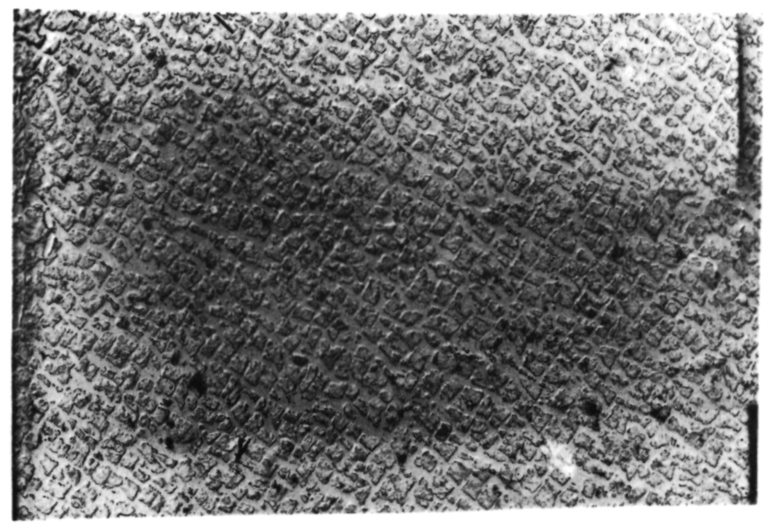
1175°C



1200°C

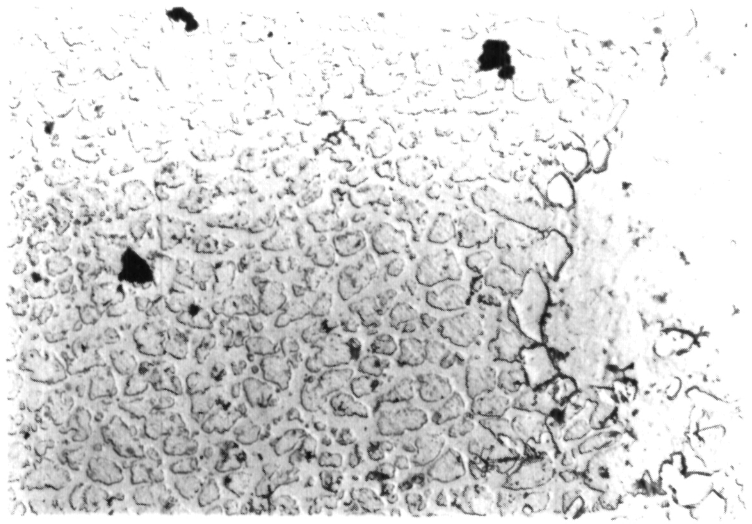


1221°C

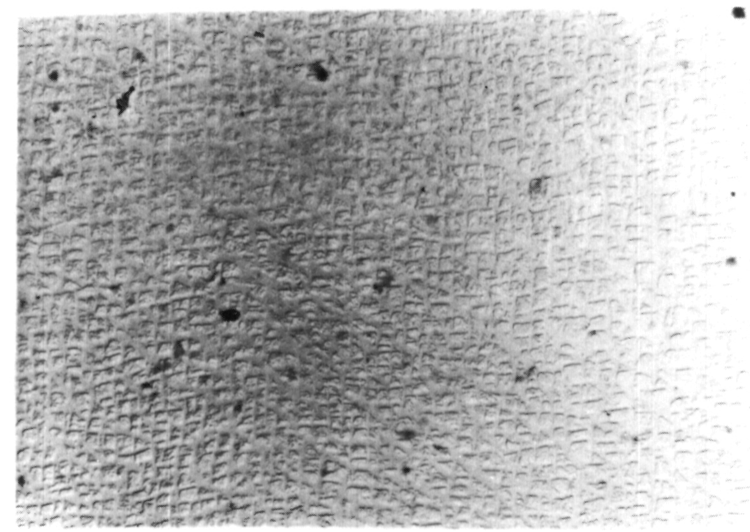


1250°C

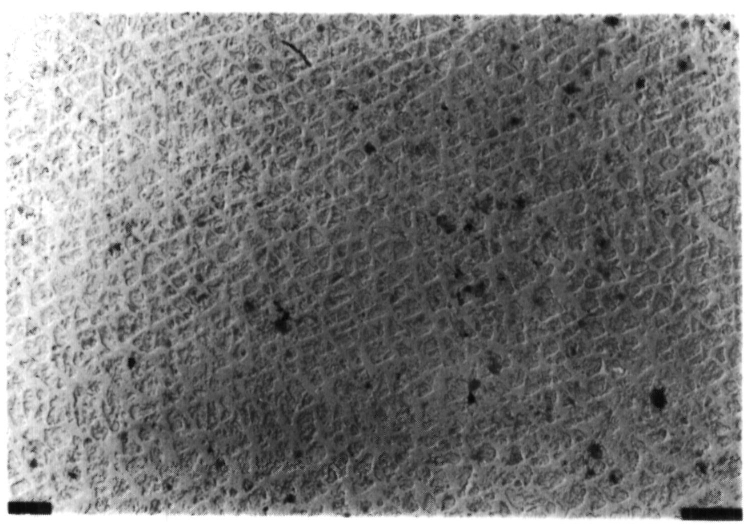
Figure 11. Transmission electron microscopy photographs of gamma prime structures in MAR-M246(Hf) at various solution heat treatment temperatures and 871°C, 10 hr precipitation heat treatment. (6000X)



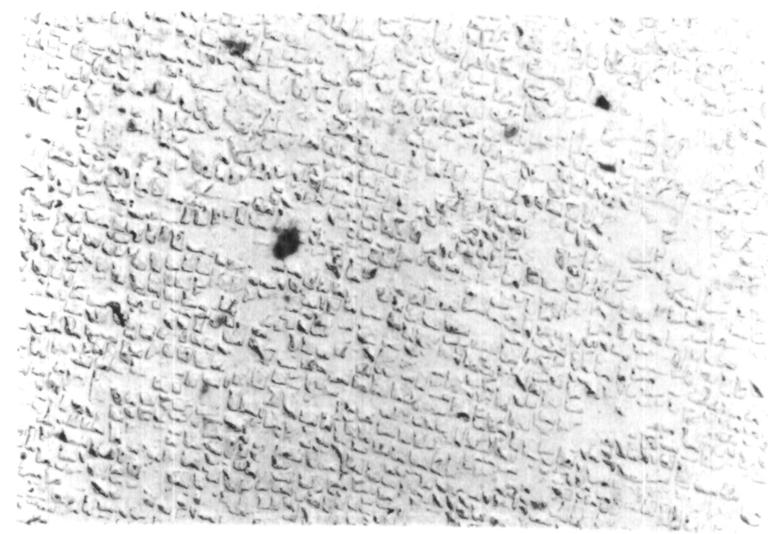
1175°C



1200°C

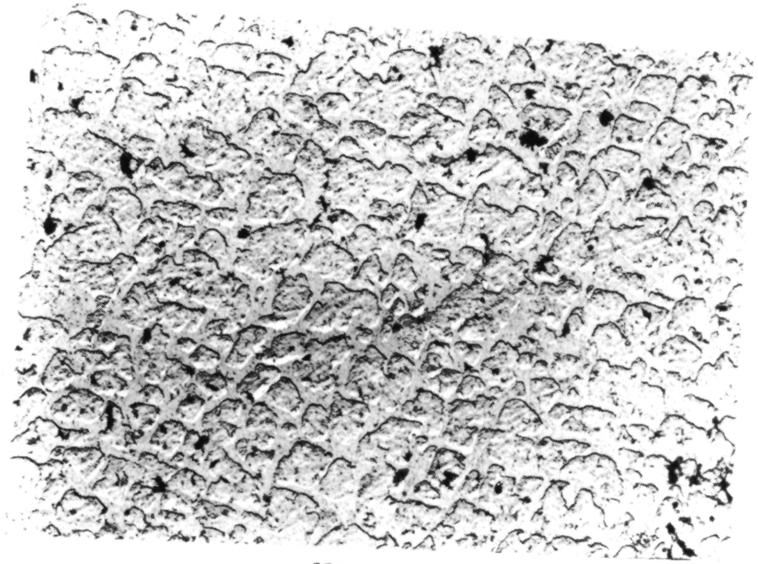


1221°C

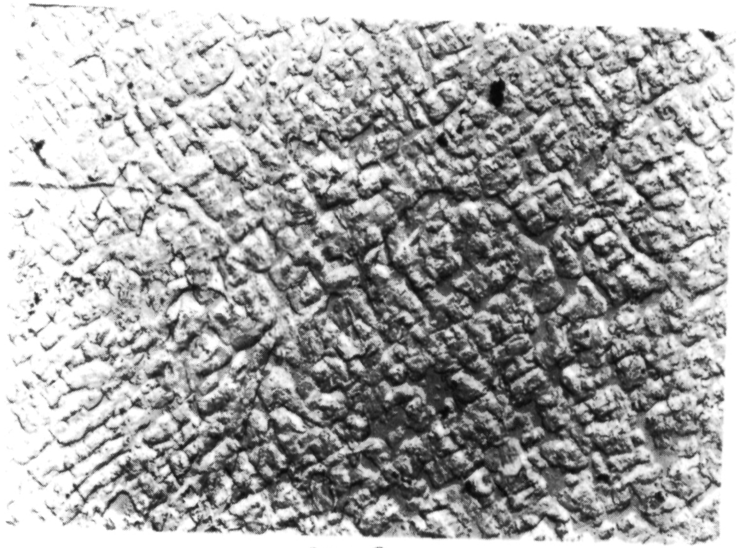


1250°C

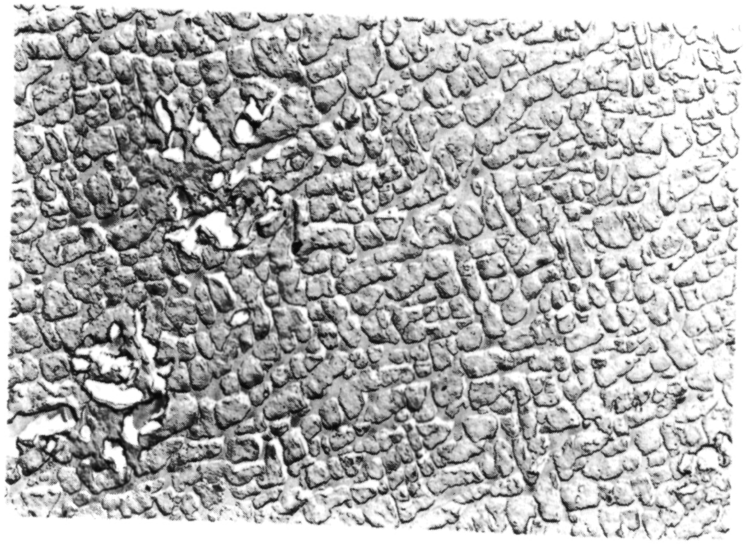
Figure 12. Transmission electron microscopy photographs of gamma prime structures in MAR-M246(Hf) at various solution heat treatment temperatures and 982°C, 24 hr precipitation heat treatment. (6000X)



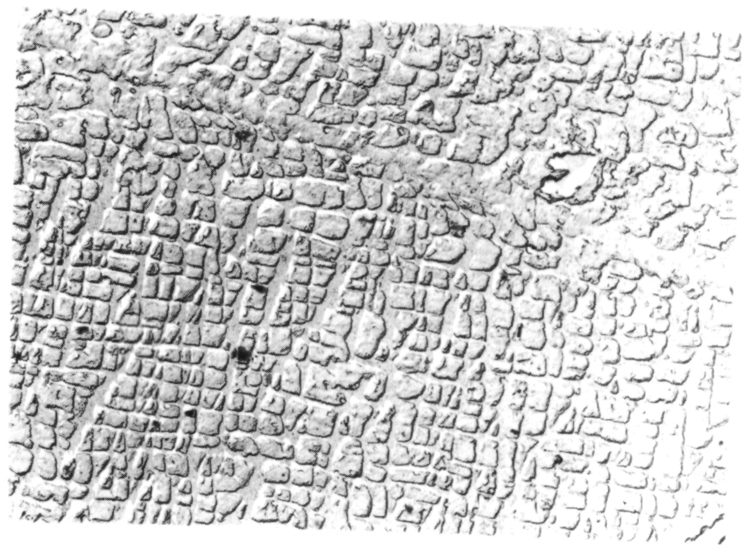
1175°C



1200°C

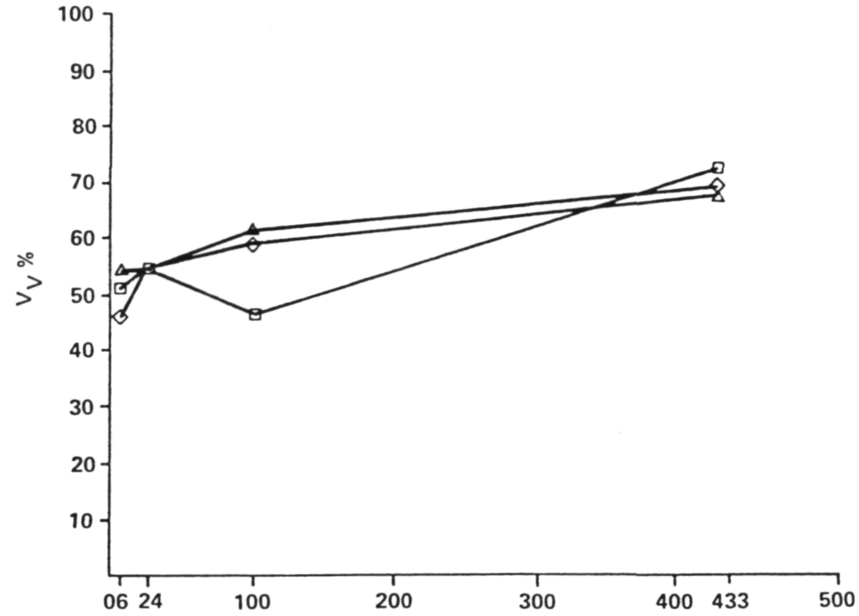


1221°C



1250°C

Figure 13. Transmission electron microscopy photographs of gamma prime structures in MAR-M246(Hf) at various solution heat treatment temperatures and 982°C, 100 hr precipitation heat treatment. (6000X)



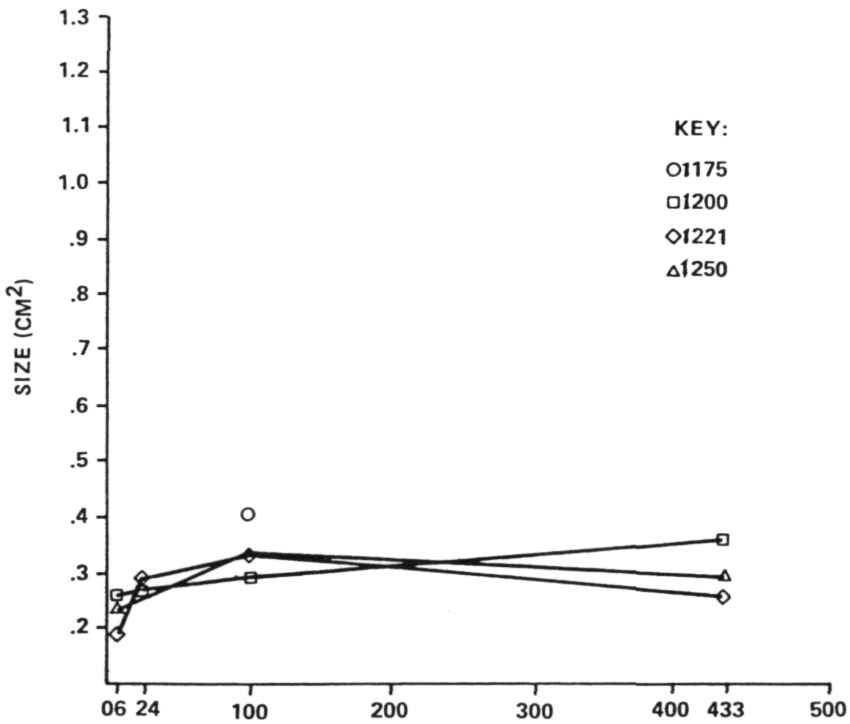


Figure 14. Size and volume fraction of γ' present as a result of heat treatment time at 871°C.

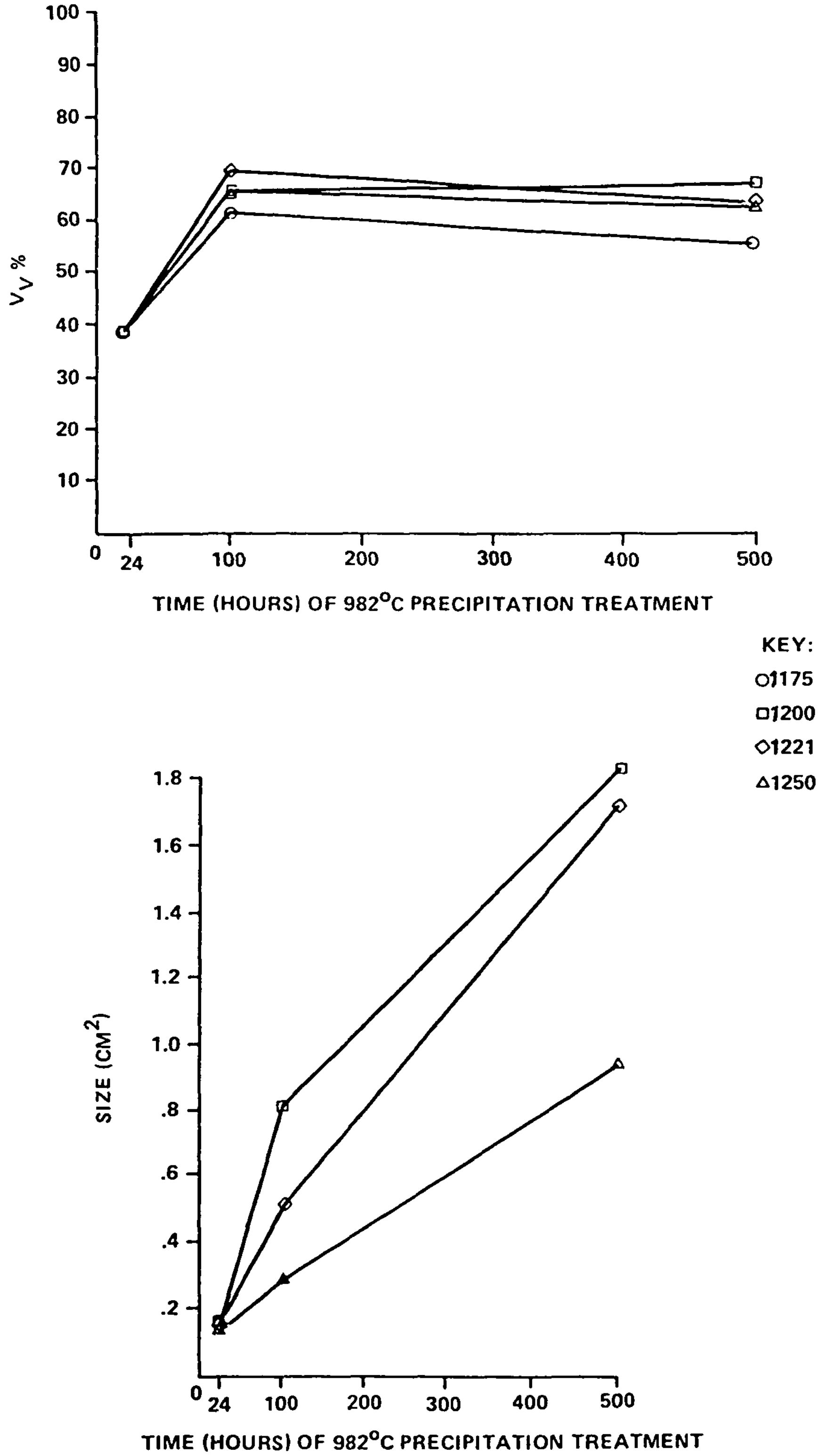


Figure 15. Size and volume fraction of γ' present as a result of heat treatment time at 982°C.

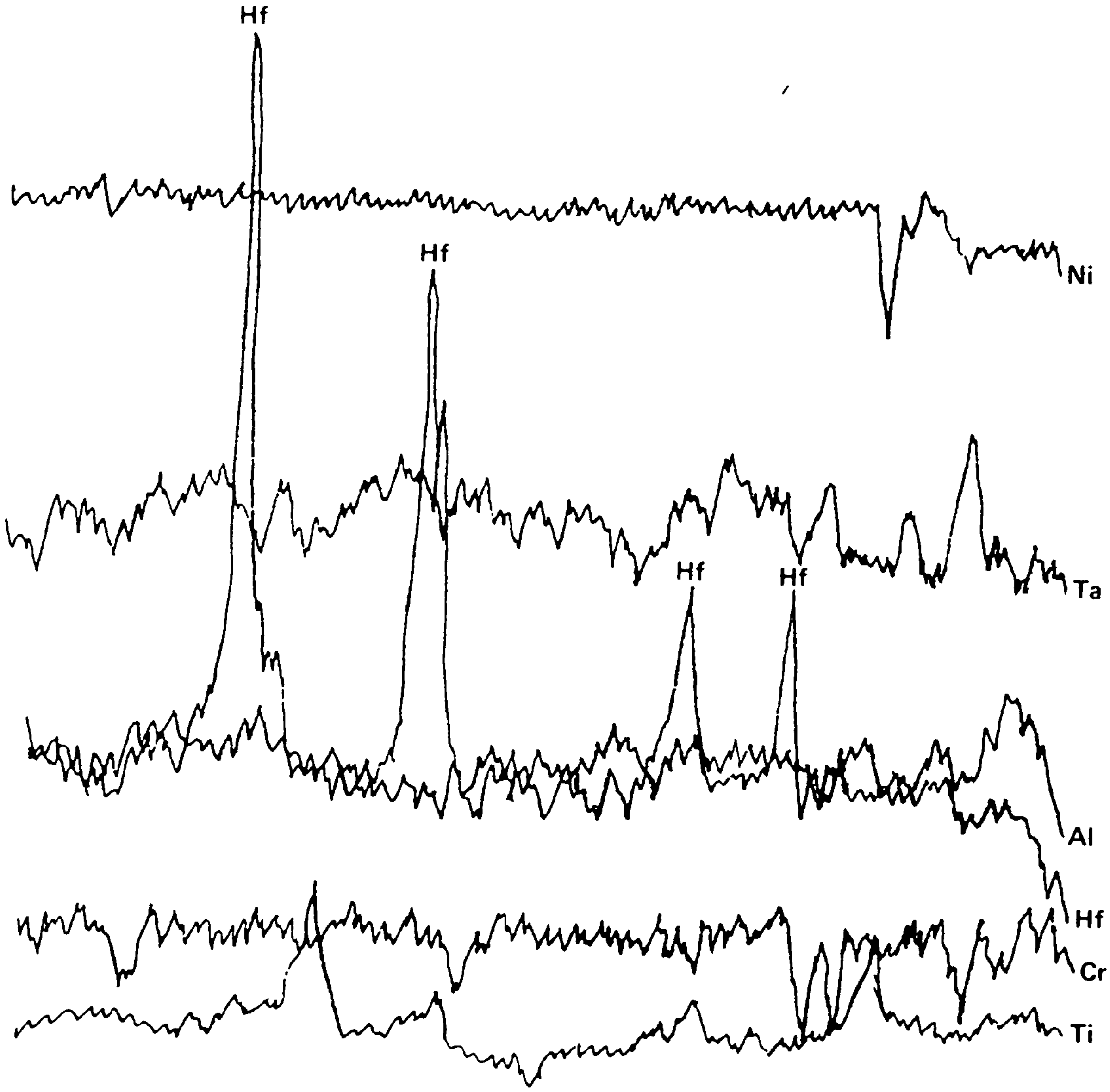


Figure 16. Microprobe trace across three secondary dendrite arms.
1221°C solution heat treatment temperature (2 hr).

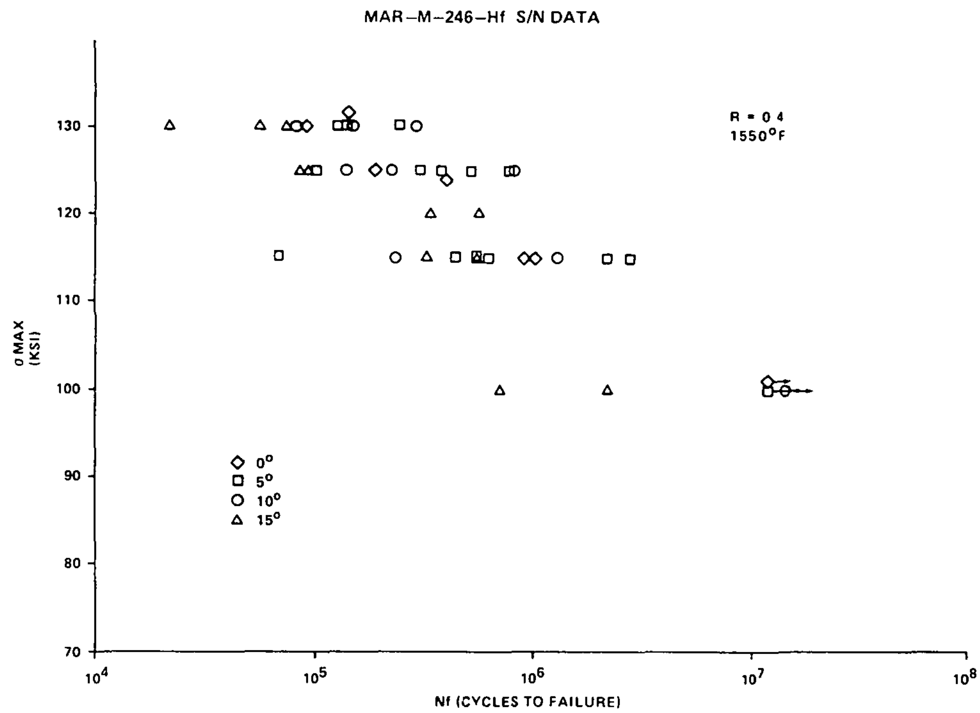


Figure 17. S/N fatigue curve, R = 0.4, 843°C.

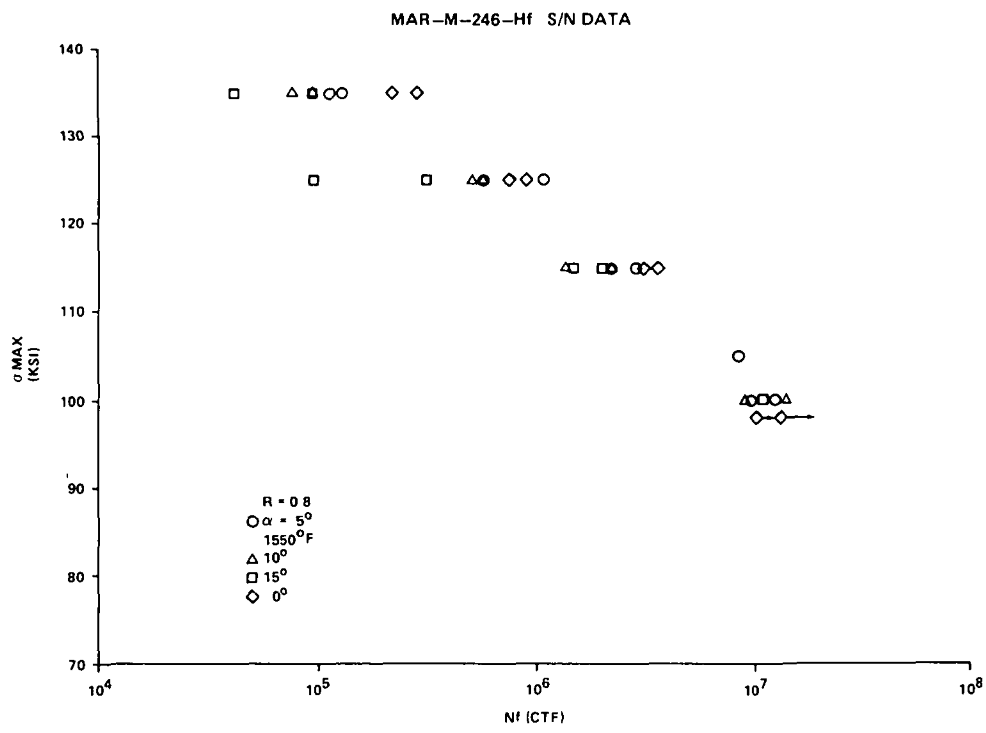


Figure 18. S/N fatigue curve, R = 0.8, 843°C.

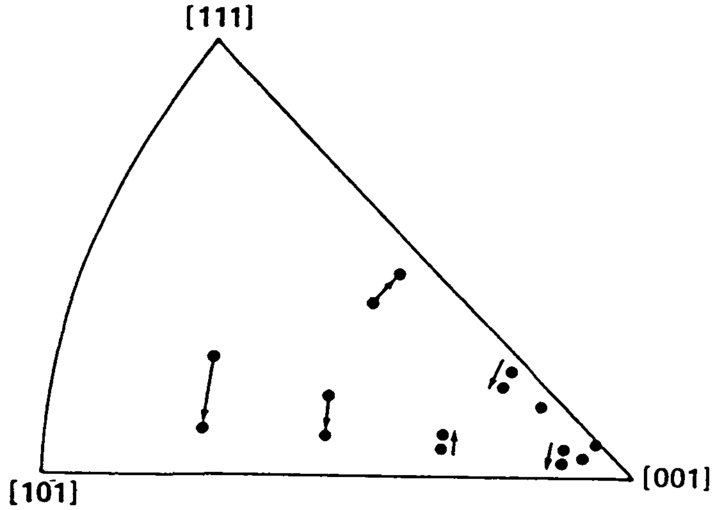


Figure 19. Crystallographic orientations of fatigue-tested specimens, showing the rotation in orientation during testing.

REFERENCES

1. Sims, Chester T. and Hagel, William C.: The Superalloys. John Wiley and Sons, New York, 1972.

2. Alter, Wendy S.: NASA TMX to be published.

3. Munafo, Paul M.: Personal communication.

4. Parr, Richard A. and Johnston, Mary H.: Met Trans A 9A, 1978, p. 1825.

5. Mackay, Rebecca A. and Maier, Ralph D.: Met Trans A 13A, 1982, p. 1747.

APPROVAL

A STUDY OF THE SOLIDIFICATION PARAMETERS INFLUENCING STRUCTURE AND PROPERTIES IN MAR-M246 (Hf)

By M. H. Johnston and R. A Parr

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

E C McKannan

R. J. SCHWINGHAMER
Director, Materials and Processes Laboratory

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